

Interlaboratory comparison of TL dating results in the Vilkiškės profile (Lithuania)

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In Poland, there are three laboratories that deal with thermoluminescence dating for geological purposes. In total, *ca.* 20,000 datings have been conducted. A small number of interlaboratory comparisons were made during over twenty years of their activity. This paper provides information on the laboratories that conducted comparative research of this kind, on the sampling places and the results. In 2001 A. Gaigalas and M. Melešytė collected 20 samples from the Vilkiškės profile in Lithuania. This profile is well known; a great deal of geological research has been made on this profile. All the 20 samples were examined at the Gdańsk TL Laboratory and 4 samples were dated at the Lublin TL Laboratory. The laboratories applied different research methods.

Keywords: TL dating, Lithuania, Vilkiškės profile, interlaboratory comparison, chronostratigraphy

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INTRODUCTION

Three out of four laboratories that existed for more than twenty years in Poland have survived until now. They are located in Gdańsk, Gliwice and Lublin.

With a total of *ca.* 20,000 samples examined, few interlaboratory comparisons have been made. In most cases, these were accidental comparisons of a small amount of datings related to three sand samples collected in Frombork. The samples were analysed in the Gdańsk and Gliwice laboratories (Fedorowicz, 1988; Bluszcz, Pazdur, 1985). The Gdańsk laboratory compared results of two loess samples from the Lower Vistula valley to those of the Kiev laboratory run by Shelkopyas (Fedorowicz, 1988, 1990, 1995; Drozdowski, 1979). The latest comparison of the Gdańsk laboratory's results concerned the profile in Maliniec near Konin. Again, the results were compared to those of the Gliwice labo-

ratory (Fedorowicz, Olszak, 1990; Stankowski, 1989). The most famous comparison was made on the loess profile in Odonów by three laboratories (in Gliwice, Lublin and Warsaw) at the end of the 1980s (Bluszcz, 1989; Butrym 1987). Results of 14 samples of loess deposits were rated very critically in this comparison; therefore, an attempt was made to identify the reasons for the discrepancies (Pazdur, 1990). The laboratories applied different methods for equivalent dose (ED) and dose rate (Dr) determination. Different methods were used to prepare samples and various grain sizes were used in measurements. Furthermore, research apparatuses and measuring techniques differed.

Interlaboratory comparison of TL dating preciseness was to be continued. However, it failed to materialize. More than 15 years have passed since the results of joint research in Odonów were published. It should be noted that during that period

major changes occurred on the map of Polish TL laboratories. The Warsaw Laboratory stopped to exist. The dating researcher in Lublin was changed. The late doctor Jerzy Butrym was replaced by Jarosław Kusiak, M. Sc. Lublin also changed its measuring methodology. At the end of the last century the author of this paper made two comparisons of the results obtained from the Chełm (Dobrowolski, Fedorowicz, Kusiak, 1995) and Siedlisko (Fedorowicz, Kusiak, Łanczont, 1997) profiles. Samples from the Lithuanian profile Vilkiškės are a third analysis of joint research.

RESEARCH PROFILE

The interlaboratory research of the Gdańsk and Lublin TL laboratories was conducted in the Vilkiškės profile. It was carried out by the author of this paper and Jarosław Kusiak from Lublin. Samples were collected by A. Gaigalas and M. Melešytė in 2001. Twenty deposit samples were collected from the profile over 37 metres high, such as samples of aeolian sands, laminated sands and fine-grained sands with humus inserts. No till samples were collected. A. Gaigalas referred the samples to chronostratigraphic units (Gaigalas, Fedorowicz, 2002).

In 2001 the Gdańsk University Laboratory conducted TL dating for all the samples collected. Its results were published (Gaigalas, Fedorowicz, 2002). In 2002, four samples used by the Gdańsk laboratory were sent to Lublin for TL dating.

MEASUREMENT METHODOLOGY

Both laboratories apply the same method of dose rate calculation. For that purpose the same apparatus is used; it is the TUKAN spectrometer in Gdańsk and the MAZAR-95 spectrometer in Lublin. A dried sample 0.5 dm³ in size is placed in the protective chamber of the spectrometer; 20 measurements are made in the time of 2000 s each (Fedorowicz, Olszak, 1985). The values of K-40, Ra-226, Th-228 are calculated as alpha, beta, and gamma radiation according to the data published by Aitken (Aitken, 1983). The above doses are adjusted for cosmic radiation. Adjustments are also made to take into account the humidity of samples in the deposit measured before the procedure.

Equivalent dose measurements are different in the two laboratories.

The Gdańsk Laboratory applies the regeneration method (Fedorowicz, 2002).

The fraction with grain size 80–100 micrometers or <50 micrometers is sifted from the entire sample. The thicker fraction is treated in 10% HCl for 1 h and then washed in distilled water only and

dried at room temperature. The silted grain fraction is ca. 3 g. Extracted grains are then divided into two unequal portions. The first, smaller portion of grains is used for the measurement of the so-called natural thermoluminescence (TN), while the remaining part is treated with an ultraviolet lamp to remove the energy stored by the sample when it was still in the deposit.

After exposure to the ultraviolet lamp, the so-called residual thermoluminescence (TL₀) is measured. It is the lowest point on the shine-down curve. After the lowest value of energy has been measured, the remaining part of extracted grains is divided into several portions. These portions are then exposed to gamma rays from a cobalt bomb until the grains regain the energy at least to the level they had in the deposit. The equivalent dose is measured with the use of a reader-analyser, model 770 A (Figs. 1 and 2). After measurements a number of verifying analyses are carried out, for

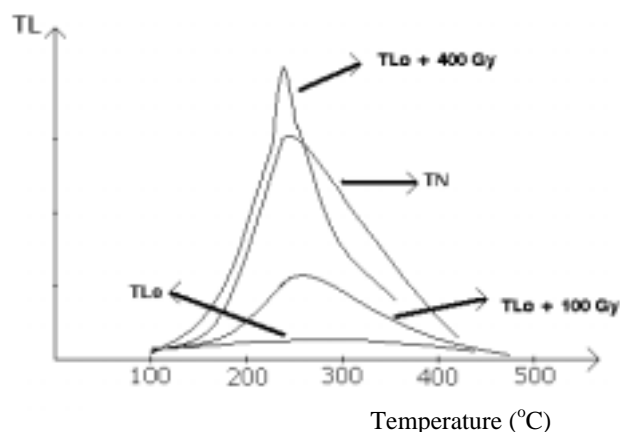


Fig. 1. TL glow curve measured for grain from the Vilkiškės profile sand sample 4 (V-4)

1 pav. Grūdelių iš Vilkiškės profilio 4-to smėlio mėginio (V-4) švytėjimo kreivės

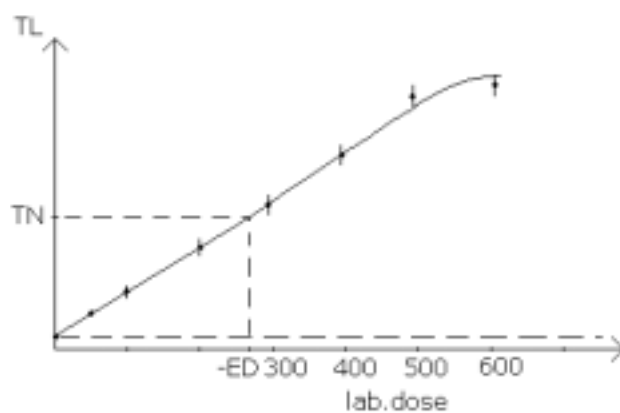


Fig. 2. Illustration of the regeneration method. Vilkiškės sand sample 18 (V-18)

2 pav. Regeneracinio metodo pavyzdys. 18-tas Vilkiškės smėlio mėginys (V-18)

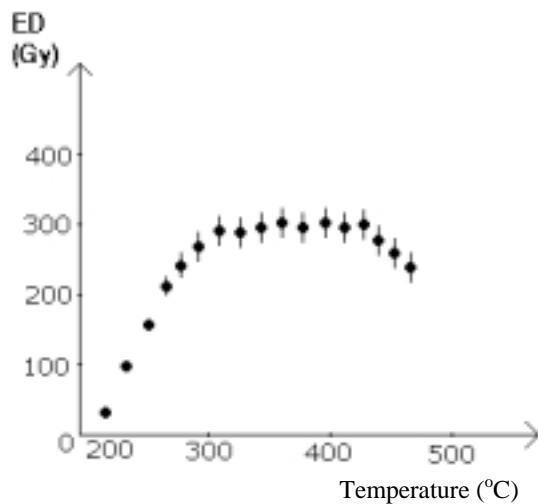


Fig. 3. An example of ED dependence on glow curve temperature exhibiting a characteristic plateau. Vilkiškės profile sand sample 18 (V-18).

3 pav. Ekvivalentinės dozės (ED) priklausomybė nuo švėtėjimo temperatūrinės kreivės, turinčios būdingą plokščią viršūnę. 18-tas Vilkiškės profilio smėlio mėginys (V-18)

example, to check for the non-linearity phenomenon and to conduct the plateau test (Fig. 3). Figure 1 exemplifies the glow curve measured for the Vilkiškės sample 4.

The Lublin Laboratory specified the equivalent dose (ED) with the partial bleach method. A polymineral fraction 45–63 micrometers in diameter was separated from each sample. It was treated in 10% HCl for an hour to remove carbonate and in 30% water solution of H_2O_2 to remove organic matter (Balescu et al., 1991).

The material was then divided into 7 parts. One part was used to measure natural thermoluminescence. The remaining six parts were exposed to ionising radiation from the source Co-60. In order to specify an equivalent dose for each part, two series of measurements were made. In the first one, thermoluminescence was measured after radiation, while in the second series thermoluminescence was measured after radiation and additionally after exposure to the ultraviolet lamp type OSRAM ULTRAVITALUX for 60 and 120 s, with 20 additional weights of 4 mg being prepared for each series.

Before measurement, mineral material was heated at a temperature of 160 °C for 5 h. Glow curves were registered with the use of the IBM-PC-compatible TL reader-analyser model RA-94 with an EMI 9789 QA photomultiplier. A BG-28 optical filter was used.

MEASUREMENT RESULTS

The results obtained by the Gdańsk Laboratory were presented in a joint paper by A. Gaigalas

Table 1. Results of the dose rate (Dr), equivalent dose (ED) and TL dating of the Vilkiškės profile samples obtained at the Lublin Laboratory (Lub) and at the Gdańsk Laboratory (UG)

1 lentelė. Vilkiškės profilio mėginių dozės rodiklio (Dr), ekvivalentinės dozės (ED) ir termoluminescencinio datavimo (TL) rezultatai, gauti Lublino (Lub) ir Gdanskos laboratorijose (UG)

Samp-les	Nr. lab.	Dr (Gy/ka)	ED (Gy)	TL age (ka BP)
V-4	Lub-4025	$1,80 \pm 0,19$	$41,0 \pm 6,0$	$23,0 \pm 4,0$
	UG-5514	$2,03 \pm 0,06$	$28,4 \pm 2,6$	$14,0 \pm 2,1$
V-5	Lub-4026	$1,65 \pm 0,13$	$36,0 \pm 2,0$	$21,0 \pm 2,0$
	UG-5515	$2,07 \pm 0,06$	$32,9 \pm 3,3$	$15,9 \pm 2,4$
V-18	Lub-4036	$1,00 \pm 0,12$	$635,0 \pm 127,0$	$632,0 \pm 145,0$
	UG-5528	$1,16 \pm 0,08$	$293,5 \pm 38,0$	$253,0 \pm 38,0$
V-20	Lub-4037	$1,73 \pm 0,15$	$767,0 \pm 107,0$	$443,0 \pm 71,0$
	UG-5530	$1,80 \pm 0,10$	$> 750,0$	$> 410,0$

and S. Fedorowicz (Gaigalas, Fedorowicz, 2002). Table 1 compares interlaboratory measurements of dose rate (Dr) and equivalent dose (ED) and TL date resulting from the division of ED by Dr.

DISCUSSION

The author of this article is a physicist. He will not discuss the potential changes in chronostratigraphy developed earlier by A. Gaigalas (Gaigalas, Fedorowicz, 2002). TL dating results for V-4 and V-5 samples fall within the range of a measuring error. In respect of the both samples, both ED and TL dates are higher at the Lublin laboratory. The Gdańsk laboratory obtained higher values for the dose rate for all the four samples under analysis.

Sample V-18 had only an approximate value of dose rate at the both laboratories. The equivalent dose was different and twice as high at the Lublin laboratory. As a result, the sample date is twice as high in Lublin. However, it is the only result that differs significantly.

The fourth sample, V-20, showed a similar result of dose rate. With the use of the regeneration method, Gdańsk was not able to specify the precise date of this sample. An indefinite date of over 440,000 years was obtained. The partial bleach method applied in Lublin allowed it to specify a precise date of this sample as 443,000 years BP.

The comparative findings presented for the Vilkiškės profile point to the need of joint interlaboratory studies. They show that application of different measurement methods may change the research result. Such results may but should not make chronostratigraphic changes in the profiles. As in the case of sample V-20, successive research methods may improve the precision of deposit dating (Table 2).

Table 2. Results of TL, OSL and ^{14}C dating of the samples of Vilkiškės profile at different laboratories
2 lentelė. Vilkiškės profilio mėginių TL, OSL ir ^{14}C datavimo rezultatai, gauti skirtingose laboratorijose

Sample No.	Depth (m)	TL age (ka) Gdańsk	TL age (ka) Lublin	OSL age (ka) Tallinn	OSL age (ka) Gliwice	C-14 age Gliwice
V-1	2,15	11,0 ± 1,6				
V-2	3,20	13,7 ± 2,0				
V-3	4,40	14,3 ± 2,1			12,7 ± 0,5	
V-4	4,50	14,0 ± 2,1	23,0 ± 4,0			
V-4a	4,60					10,8 ± 0,16 7,7 ± 0,16 10,3 ± 0,1
V-5	4,70	15,9 ± 2,4	21,0 ± 21,0			
V-6	5,20	18,3 ± 2,7				
V-7	12,45	21,4 ± 4,9				
V-8	13,50	34,8 ± 5,2				
V-9	14,50	37,2 ± 5,6			129,0 ± 24	
V-10	15,50	38,0 ± 5,7				
V-11	17,80	54,8 ± 8,2			150,7 ± 5,8	
V-12	19,50	66,6 ± 10,0				
V-13	21,05	103,0 ± 15,0				
V-14	22,40	192,0 ± 28,8				
V-15	24,10	182,3 ± 28,0				
V-16	25,70	184,9 ± 28,0			211,0 ± 16	
V-17	27,00	188,4 ± 28,3				
V-17a	27,50			250 ± 17,4		
V-18	28,60	253,0 ± 38,0	623,0 ± 145,0	>250,0		
V-19	33,00	280,6 ± 42,0		>250,0	127,9 ± 6,3	
V-20	33,75	>410,0	443,0 ± 71,0	>250,0		

Despite the fact that the luminescent methods have been used in geological research for nearly 30 years, they constantly develop. Any development of measuring methods improves the precision of measurements. Interlaboratory comparative studies should give an impetus to their further development.

This paper was written thanks to the work and personal involvement of Jarosław Kusiak from the Physical Geography Department of Maria Skłodowska-Curie University. The author wishes to express his gratitude for co-operation.

CONCLUSIONS

Interlaboratory comparisons of 4 samples gave different results. The results of two first samples (V-4 and V-5) fall within acceptable tolerances. They do not cause any changes in the Late Glacial table. The third sample (V-18) gave unexpectedly different results. It is difficult to discuss these differences. The fourth sample (V-18) allowed the determination of a finite date, while the Gdańsk laboratory obtained an indefinite date.

It should be borne in mind that interlaboratory research reveals dating differences. To compare the results on the profiles analyzed, one should use the

data of one laboratory. Interlaboratory research should not be neglected. It should be encouraged and supported. It contributes to the development of methodology and natural sciences.

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VILKIŠKĖS ATODANGOS (LIETUVA) NUOGULŲ TL DATAVIMO REZULTATŲ TARPLABORATORINIS PALYGINIMAS

S a n t r a u k a

Lenkijoje veikia trys laboratorijos, kuriose atliekamas termoluminescencinis (TL) datavimas geologiniams tikslams. Šiuo metu yra atlikta apie 20 000 datavimų. Tačiau daugiau negu per dvidešimtį šių laboratorijų veiklos metų kontrolinių tarplaboratorinių datavimų nėra daug. Jų rezultatai paskelbti mokslinėje spaudoje.

2001 m. surinkti mėginiai iš Vilkiškės atodangos Rytų Lietuvoje buvo datuoti Gdanskio universiteto Termoluminescencijos laboratorijoje (iš viso 20). Keturi kontroliniai datavimai atlikti Liublino universiteto laboratorijoje. Šių minėtų laboratorijų tyrimo metodai šiek tiek skyrėsi. Straipsnyje aprašyta tyrimų metodika, naudota tarplaboratoriniam duomenų palyginimui, kontrolinio datavimo rezultatai.

Станислав Федорович

МЕЖЛАБОРАТОРНОЕ СРАВНЕНИЕ РЕЗУЛЬТАТОВ TL ПРОФИЛЯ ВИЛЬКИШКЕС (ЛИТВА)

Р е з ю м е

В Польше три лаборатории занимаются термолуминовым датированием (TL) для геологических целей. Уже сделано около 20 000 датирований. За выше двадцати лет их деятельности осуществлено небольшое количество межлабораторных сравнительных работ. Которые лаборатории осуществили эти исследования, откуда происходили исследованные образцы, какие получены данные в результате проведенных испытаний – ответы на вопросы подобного рода можно найти в настоящей публикации.

В 2001 г. А. Гайгалас и М. Мелешите отобрали 20 образцов из профиля Вилькишкес в Литве. Для этого общеизвестного профиля выполнено много исследовательских работ. Упомянутые 20 образцов были исследованы в лаборатории TL в Гданьске, а четыре из них датированы в лаборатории в Люблине. Методы исследования – разные.

В статье представлены методика, использованная в этих сравнительных исследованиях, а также результаты сравнений.